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EFFECT OF MICROSTRUCTURE AND HARDNESS PROPERTIES OF AL2011 BASED COMPOSITES FOR TITANIUM DI BORIDE USING STIR CASTING METHOD

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Original Article

ABSTRACT

Objectives

Al-2011 base composite reinforced with TiB_2 salts was successfully manufactured by the stir-casting technique. Conversion of the matrix with the inclusion of copper and titanium improved the mechanical properties. The analysis is attempted with the proportion of Al2011+K_2TiF_6+KBF_4 using stir casting process, microstructures and hardness test is done. SEM-EDX and XRD investigation expose existence of fine TiB_2 particles in before and after heat treatment both as cast and extruded condition.

Methods/Statistical Analysis

Aluminum 2011 material is fabricated by stir casting method with added titanium diboride on various weight percentage compositions. After, the developed composites were subjected to extrusion appearing in a heat of 400^o C applying a 50 T hydraulic hit. Scanning electron microscopy-energy dispersive X-ray analysis (SEM-EDX) and X-ray diffraction (XRD) systems tests were carried out on as cast and extruded metal matrix composites. The investigation of microstructure of aluminum titanium diboride metal matrix composites with specific attention on the before, after as cast and extrusion behavior of the composites.

Findings

It is detected that, the couple as cast and extruded compound do exhibit significant grain refinement. Mean while after the secondary process as per the observation the grain reinforcement of the extruded composites reveal significant of 5%, 10% opposing the as cast samples. After raising the metal matrix composites to 10% was plastically deranged shows the value 171M Pa. The SEM of the extruded samples shows enlarged grains of aluminum solid solution with intermetallic particles at the grain boundaries to restrict the deformation. This result improves the extrusion process heat treated samples well in grain enlightening and uniform particle distribution compared to as cast condition of samples. To compare the 5%, 10% as cast samples the plastic deformation process gives the 10% extruded heat treated samples with higher strength 84 to 90 HV.

Application/Improvements

It used in multiple industrial applications like aerospace, marine, automotive, rail, building, packaging, energy distribution, sports and leisure, mechanical industries and engineering, etc. The nature of Al-2011 with TiB2 are lightness, corrosion resistance, suitability for surface treatments, the diversity of the alloys and intermediates, ease of use, Recycling, electrical and thermal conductivity, etc. When a compared with as cast matrix alloy and its composite,

extruded alloy and its composite exhibits higher extent of grain refinement.

KEYWORDS: Aluminium Alloy, Potassium Hexafluro Titanate, Potassium Tetrafluro Borate, Hardness

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1. INTRODUCTION

Stir fabricated composites which are low in cost for fabrication. The salts like potassium hexafluro titanate (K_2TiF_6) and potassium tetrafluro borate (KBF_4) mixed with aluminum 2011 prepared by stir casting method are the properties used to improve MMCs. The direct extrusion process of reinforcement with the TiB_2 particles with aluminum alloy boost the grains in the matrix and raise the interfacial fixing strength of the composites. In the analysis the tensile properties of heat treated as cast and extruded Al2011-10wt% TiB_2 composites have higher strength compare to the Al2011-5wt%TiB_2 composites. The content of TiB_2 particles with aluminium metal matrix increases the strength in both as cast and extruded conditions are observed. Among the secondary processing routes the extrusion is emerged as an important route owing to its ability to produce high strength corrosion resistance components within a short duration. TiB_2 is the most stable of several titanium-boron compounds. The material does not occur in nature but may be synthesised by carbothermal reduction of TiO_2 and B_2O_3. TiB_2 is resistant to oxidation in air up to 1000°C. It is also resistant to HCl and HF but reacts with H_2SO_4 and HNO_3. It is readily attacked by alkalis. In combination with other primarily oxide ceramics, TiB_2 is used to constitute composite materials in which the presence of the material serves to increase strength and fracture toughness of the matrix. The corrosion resistances of aluminum metal matrix composites are improved by the secondary process (Ramesh, 2011).

2. EXPERIMENTAL DETAILS

In this part, matrix material, reinforcement, experimental setup and technicality of compositions of composites are considered.

2.1 Matrix and Reinforcement Used

In this work aluminum 2011 alloy is taken as matrix metal and titanium di boride is taken as reinforcement. Their properties are provided in Table-1.

Table 1: Physical and Mechanical Properties of Al-2011 and TiB_2

Properties	Aluminium 2011	Titanium di Boride
Density(gm/cm^3)	2.82	4.53
Tensile Strength(MPa)	365	410-448
Coefficient of Thermal Expansion($10^{-6}/^oC$)	23	15
Modulus of Elasticity(GPa)	70-80	510-575
Poission's Ratio	0.33	0.1-0.15
Melting Point(^oC)	535	2970
Hardness Vickers(HV)	110	1800

2.2. Composite Preparation and Extrusion

In Al2011-TiB_2 composite was fabricated by stir casting technique owing to its simplicity and low cost. The salts like potassium hexafluro titanate (K_2TiF_6) and potassium tetrafluro borate (KBF_4) mixed with aluminum 2011 composites.

A mixture of 2.82 kgs of Al 2011 metal along 5wt% of TiB_2 and 5.64 kgs of Al 2011 metal along 10wt% of TiB_2 adept metals were melted in the heater to a heat of 800^oC. Details of stir casting reaction and TiB_2 formation are discussed elsewhere. The molten alloys are mixed by using mechanical stirrer, rotates at a speed of 700 rpm for duration of 15 minutes. The melted composites are maintained at a temperature of 700- 800^oC and poured in to metallic moulds. The percentages of TiB_2 formation in the composite are predicted by dissolving the known quantity of sample in hydrochloric acid. The matrixes are dissolved in the acid by leaving TiB_2 and Al_3Ti particles. The obtained particles were cleaned and dried for weighing samples of composites as shown in Figure 1.

Figure 1: Samples of Composite Containing Weight % TiB_2 in Aluminium2011 Matrix

The percentage of TiB_2 and Al_3Ti particles are estimated by considering the difference in the weight of the composite and obtained particles. The percentage of TiB_2 in the extracted particles were consistent by comparing the ratios of high intensities of Al, Ti and Cu extracted from SEM-EDX, Al,TiB_2 and Al_3Ti particles obtained from XRD studies. These powders of stotchiometric composition based on equation (1)

$$2Ti+2B+3Al \rightarrow TiB_2+Al_3Ti \quad (1)$$

The overall reaction showing the formation of TiB_2 can be writtern as,

$$K_2TiF_6(1)+KBF_4(1)+Al(1) \rightarrow TiB_{2(in\ Alloy)}+KFAlF_3(1)+KF(1)+AlF_3(1) \quad (2)$$

The synthesis of stir Al-TiB_2 can also carried out from the Al-TiO_2-B and Al-TiO_2-B_2O_3 systems. Using Al-TiO_2-B system types of reinforcement, i.e. Al_2O_3, TiB_2 and Al_3Ti can be created during this process. The equations for chemical reactions are:

$$3TiO_2+13Al \rightarrow 2Al_2O_3+3Al_3Ti \quad (3)$$

$$3TiO_2+6B+4Al \rightarrow 2Al_2O_3+3TiB_2 \quad (4)$$

From the above reaction we can summarized that the amount of Al_2O_3, TiB_2 and Al_3Ti can be control by varying the B content. Using Al-TiO_2-B_2O_3 system the following chemical reactions are:

$$B_2O_3+2Al \rightarrow Al_2O_3+2B \quad (5)$$

$$3TiO_2+4Al \rightarrow 2Al_2O_3+3Ti \quad (6)$$

It is proposed that, Ti can also react with Al and B to form titanium aluminum borides $(Ti,Al)_x B_y$ by following reaction :

$xTi + x\,Al + yB \rightarrow \{Ti,Al\}_x B_y$ (7)

Studies reveal that the primary TiB_2 particles on the surface of $TiAl_3$ are appreciably free and movable and because of boron diffusion over boundary layer towards $TiAl_{3,}$ TiB_2 Particles produces during growth with the primary ones formed agglomeration ring. This can be expressed as

$TiAl_3+2B+\rightarrow TiB_2+3Al$ (8)

The production of TiB_2 ceramic particles in the stir casting process are shown below,

$Ti+3Al\rightarrow Al_3Ti$ (9)

$Ti+2B \rightarrow TiB_2$ (10)

$Ti+2B+3Al\rightarrow TiB_2+Al_3Ti$ (11)

In the secondary process of the direct extrusion the molybdenum disulphide type of gel and 50mm diameter to 500mm length billet sizes cover range is used. Then it is pre heated at the temperature of 700^{o}C.[9] The cast of Al2011 with TiB_2 composites were deal with direct extrusion at a working heat of 400^{o}C using 50T hydraulic hit at the pressure range 25 to 50 tons are done in M/s Forging and allied products private Limited, Bangalore, Karnataka, India. The composites were produced at the range of 15mm diameter and 125mm height. The extrusion ratio of 15 was adopted for both 5% and 10% composite with the constant extrusion speed of 0.6mm/sec (Zhangwei Wang, 2010).

2.3. Heat Treatment of Al2011-TiB_2 composites

After the experiment for this study, as cast and extruded Al2011-TiB_2 composites bar of sizes 15 x 15 x 5 mm, 110 mm length x 12.5mm diameter was heat treated at 400^{0}C for 48 hours followed by air cooling. Then the samples are finally polished at the size of 10x10x2.5 mm and 100mm length x 10mm diameter were cut from the bar and polished by the paper of silicon carbide grit size ranging from 500 and 1000. Before drying the samples in warm air, they are cleaned with distilled water and acetone (Srivastava, 2007).

2.4. SEM-EDX Measurement

Both as cast and extruded composites were deal with optical microstructure studies. The SEM-EDX specimens (waterless filter paper) were cut in 10x10x2.5mm size as shown in Figure 2.

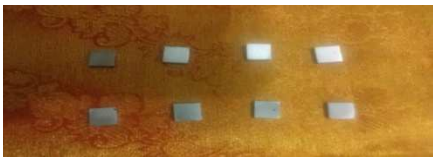


Figure 2: SEM and EDX Test Samples

For electrically conductive vacuum coating unit very thin film of gold and palladium deposited on the surface of the sample are used. Fine coating was done by evaporating Au-Pd plate under inert atmosphere (argon environment). The SEM- EDX analyses were done by computer controlled field emission SEM (JEOL JSM-6330F, JEOL Ltd., Akishima Tokyo 196-8558 Japan) equipped with a EVEX-EDX detection system, Princeton, Gama Tech Instruments, NJ, 8540, US. In the present investigation, the SEM was used in common and emissive mode. The current density was restricted to 5.0 keV to reduce the contribution of silicon substrate and minimize the loss of re- grown aggregates are investigated

(Bob Hafner, 2015).

2.5. X-Ray Diffraction Analysis

Philips, X'Pert PRO X-ray diffraction system, PANalytical, Holland, are the XRD analysis with a curved position sensitive detector (PSD) which gives a resolution of 0.03 mm over a range of 10° - 80, 2θ using standard techniques.

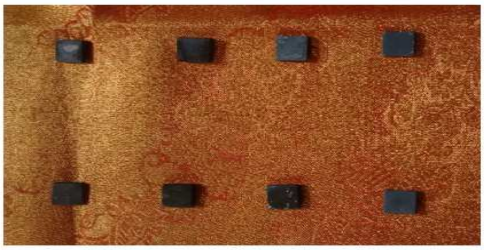


Figure 3: XRD Test for Corrosion Tested Samples

In refection geometry (using Cu Kα1 radiation), with the tube operating at 40 kV and 30 mA and with a beam size of 10mm were measured. Sample 10x10x2.5 mm Figure 3 shows (waterless filter paper) are pasted on a metal slide. At the rate of 2° per minute for a period over 3.1750 sec to ensure smooth diagnostic peaks the data's are collected (Cressey, 1996).

2.6. Micro Hardness Measurement

The shape of pyramidal diamond indenter that forms a square indent is in Vickers micro ranges. To find the progress of hardness in the fabricated Al- 5%wt TiB_2, Al-10%wt TiB_2 composite micro hardness test was done at a load of 5 kg with 15 seconds dwell time and the specimen processed for micro-hardness as shown in Figures 4, 5.

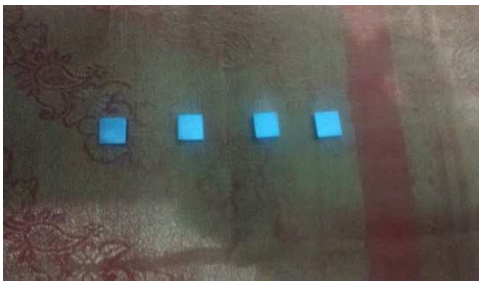


Figure 4: Hardness Test for As Cast Samples (Al-5%TiB_2 & Al-10%TiB_2)

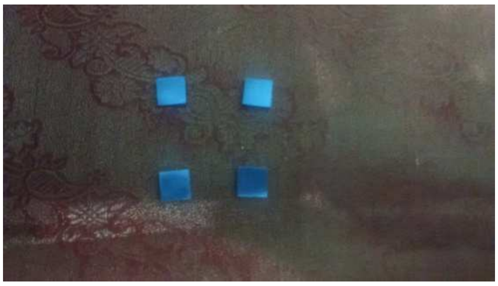


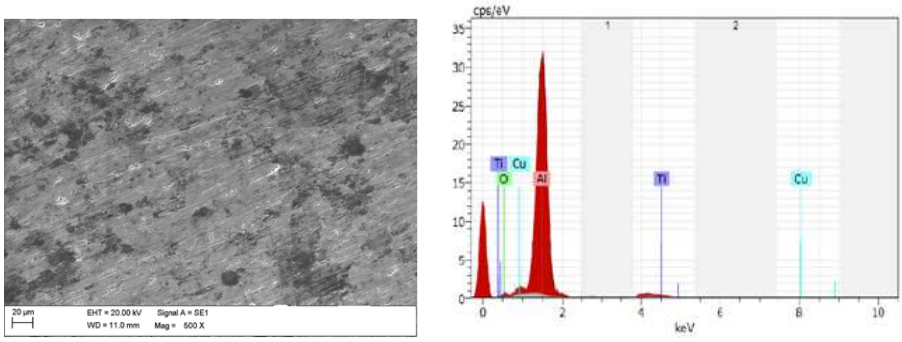
Figure 5: Hardness Test for Extrusion Samples (Al-5%TiB_2 & Al-10%TiB_2)

The shape of the specimen is 10x10x2.5mm for the experiment. The test samples were polished before performing micro hardness test by using belt grinder and disc polisher (Keneth Kanayo Alanemea, 2013).

3. RESULT AND DISCUSSIONS

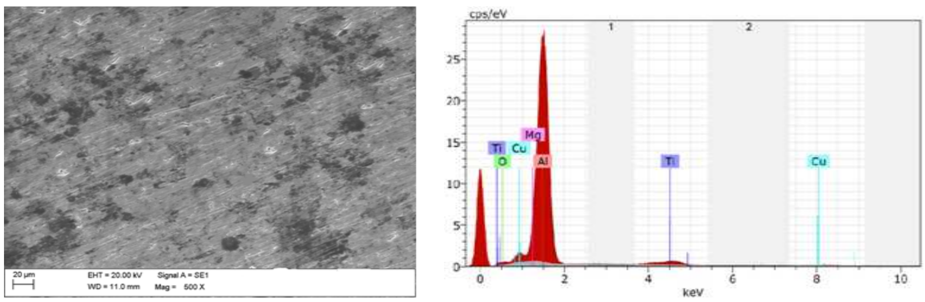
3.1. SEM-EDX of Al2011-TiB_2 Composite

EDX is an analytical technique used for elemental composition analysis of the composites. There is not seen any reaction product between the Al2011–TiB_2 interfaces. EDX analysis the hardness of the aluminum is improved by adding TiB_2 particles. SEM analysis the size of the TiB_2 particulates decreased with increasing Al contents in the blends. Composite shows that the 10Wt% TiB_2 interfacial bonding is better as compared to the 5Wt% TiB_2 proportions. Cps/Kev denotes the counts per second per electron-volt.



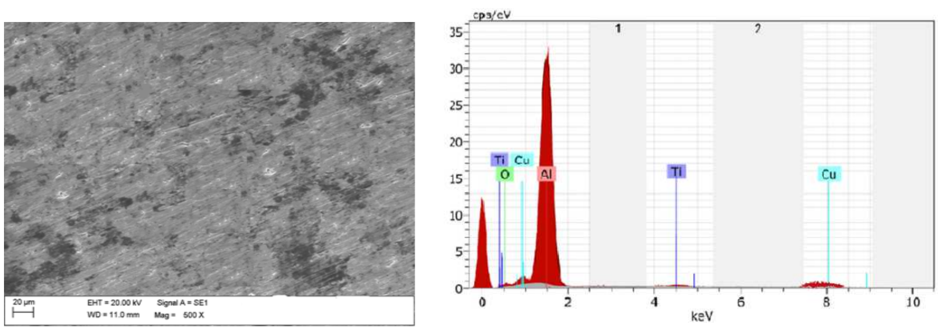
(a) Surface Morphology through SEM **(b) EDX**

Figure 6: Sample A: EDX of Al–2011 with 5wt%TiB_2 (As Cast before Heat Treatment)



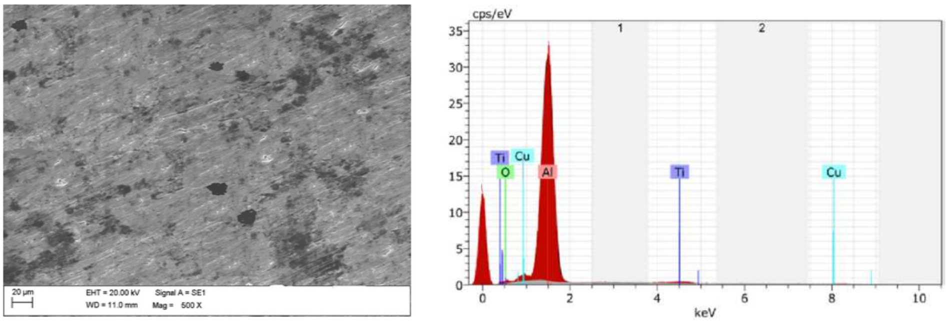
(a) Surface Morphology through SEM **(b) EDX**

Figure 7: Sample B: Al–2011 with 5wt%TiB_2 (As Cast after Heat Treatment)



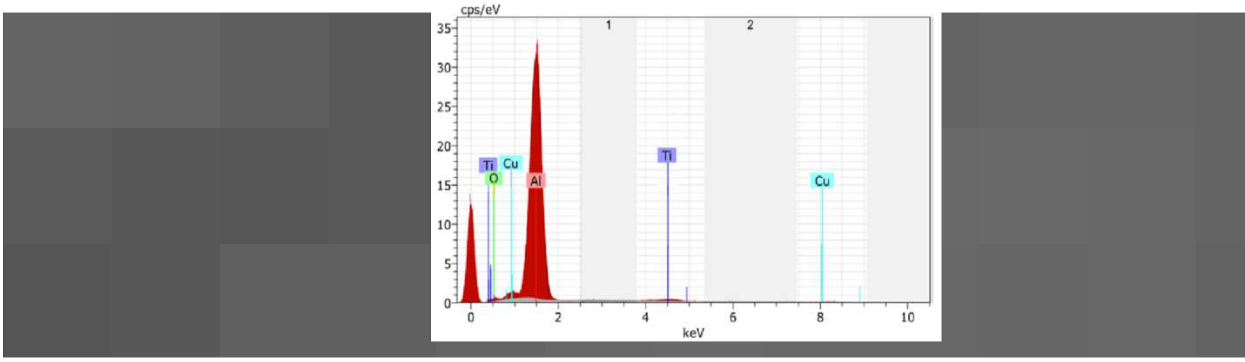
(a) Surface Morphology through SEM **(b) EDX**

Figure 8: Sample C: Al–2011 with 10wt%TiB_2 (As Cast before Heat Treatment)



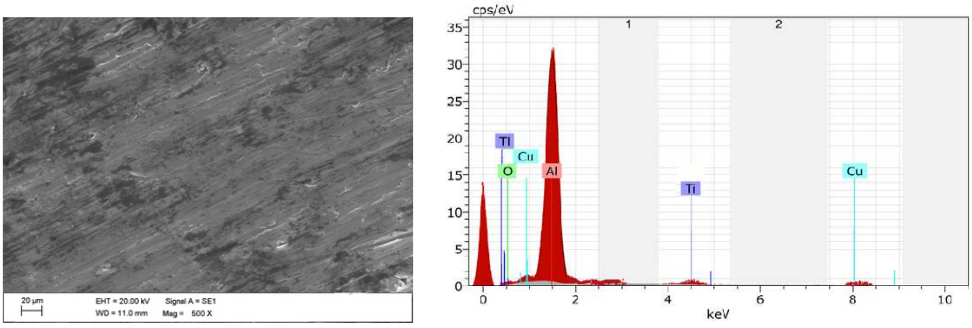
(a) Surface Morphology through SEM (b) EDX

Figure 9: Sample D: Al–2011 with 10wt%TiB$_2$ (As Cast after Heat Treatment)



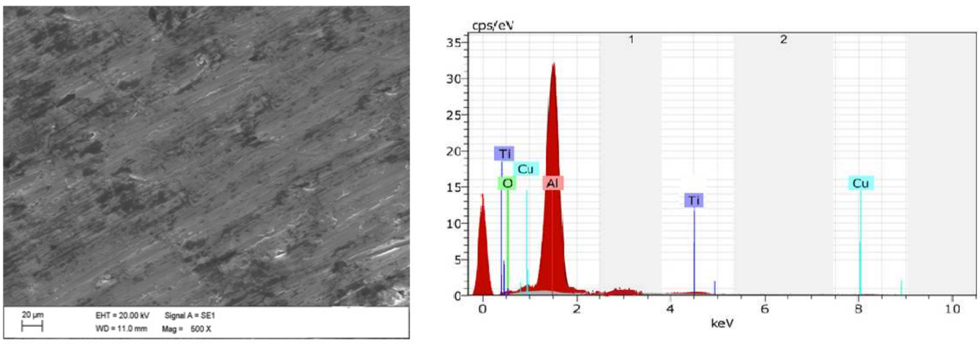
(a) Surface Morphology through SEM (b) EDX

Figure 10: Sample E: Al–2011 with 5wt%TiB$_2$ (Extruded before Heat Treatment)



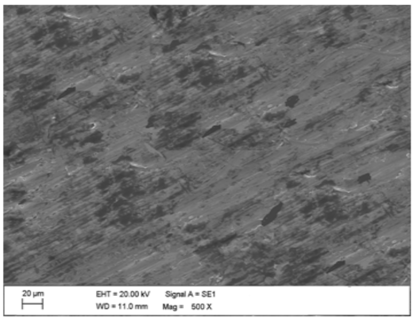
(a) Surface Morphology through SEM (b) EDX

Figure 11: Sample F: EDX of Al–2011 with 5wt%TiB$_2$ (Extruded after Heat Treatment)

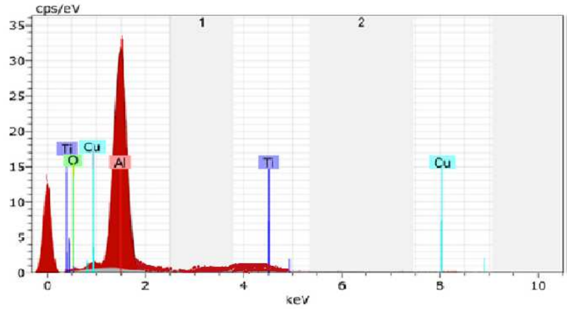


(a) Surface Morphology through SEM (b) EDX

Figure 12: Sample G: EDX of Al–2011 with 10wt%TiB$_2$ (Extruded after Heat Treatment)



(a) Surface Morphology through SEM



(b) EDX

Figure 13: Sample H: EDX of Al–2011 with 10wt%TiB_2 (Extruded after Heat Treatment)

The figures 6,7,10,11, shows that Al–2011 with 5wt%TiB_2 aluminum counts gradually reduced and also the reinforcement particle increased. The reinforcement particles sizes are less than 1µm. To know the presence of TiB_2 particles the quantitative elemental analysis are performed by EDX testing. The Figures 8,9,12,13 shows that Al–2011 with 10wt%TiB_2 aluminum counts gradually increased and also the reinforcement particle increased. EDX spectrum of the Al-TiB_2 10% extruded after heat treatment composites had better hardness compare to the as cast composites.

In EDX analysis Y-axis represents the counts of X rays received. The revealer and X- axis represents energy level of those no of counts value in Table 2.

Table 2: % Weight of Different Elements of Al2011 with TiB_2 Metal Matrix Composites

Process	AL2011 + % of TiB_2	BH=Before Heat Treatment AH=After Heat Treatment	Sample Code	Element	Series	Unn (wt%)	Norm c (wt%)	Atom c (at%)	Error (3Sigma)
Cast	5%	BH	A	Aluminum	K	87.20	88.07	86.20	12.58
				oxygen		6.74	6.81	11.23	3.92
				Titanium		3.19	3.22	1.77	0.41
				copper		1.88	1.90	0.79	0.33
		AH	B	Aluminum	K	88.01	88.05	87.91	10.42
				oxygen		5.40	5.78	6.45	3.82
				Titanium		2.12	2.45	2.12	0.31
				copper		1.62	1.14	1.48	0.93
	10%	BH	C	Aluminum	L	85.01	84.22	85.31	10.58
				oxygen		5.45	5.25	5.40	3.72
				Titanium		2.92	2.72	2.82	0.41
				copper		1.62	1.64	1.68	0.33
		AH	D	Aluminum	K	89.01	89.43	88.01	10.54
				oxygen		5.46	5.25	5.43	2.72
				Titanium		4.12	3.12	3.12	0.51
				copper		0.95	1.98	1.62	0.33
Extruded	5%	BH	E	Aluminum	L	85.45	85.042	85.01	14.58
				oxygen		6.95	6.45	6.45	3.92
				Titanium		3.61	3.17	3.42	0.48
				copper		2.62	2.65	2.58	0.43
		AH	F	Aluminum	K	89.04	89.45	89.81	10.54
				oxygen		4.27	4.73	4.45	2.92
				Titanium		5.42	5.12	5.22	0.41
				copper		1.78	1.45	1.21	0.33

	10%	BH	G	Aluminum	K	88.45	88.01	88.05	11.52
				oxygen		6.85	6.42	6.43	3.92
				Titanium		3.45	3.62	3.42	0.71
				copper		1.67	1.58	1.23	0.47
		AH	H	Aluminum	K	91.24	91.21	90.01	7.52
				oxygen		2.15	3.05	4.05	1.92
				Titanium		5.82	5.12	5.82	0.20
				copper		2.01	2.08	1.68	0.14

Moseley's Law is basis for the EDX analysis. Moseley's Law was shown in the equation 12.

$$E = C_1 (Z - C_2)^2 \tag{12}$$

Where E= energy of the emission line

Z= atomic number of the emitter

C_1 and C_2 are constants (Tarek, 2009).

3.2. XRD of Al2011-TiB_2 Composite

The figures 14,15,16,17,18,19,20,21 shows that the XRD pattern of extracted powder consisting of TiB_2 and Al_3Ti particles. The estimated percentage of TiB_2 particles from the ratio of peak intensity in XRD pattern is found to be 4.9% approximately. The figure shows the optical microphotographs of both as cast and extruded Al2011-TiB_2 composites had peak strength of the aluminum proposition. The microstructure shows uniform distribution of TiB_2 particles in the matrix material. By heating the Al–Ti–B system to 1000˚ C and 900˚C the analytical work of Al_3Ti begins to appear. Until TiB_2 was formed at 570°C to 595°C, Al_3Ti increases continuously. In the current work, the mixture was introduced at 700˚C - 800˚C. Al_3Ti was likely formed, before the remaining Ti reacts with the formation of desired TiB_2. This explains the increased intensity and coarsening of Al_3Ti flakes with increased volume fractions as described earlier. The sequential overall reactions for the formation of TiB_2 should thus be:

$$Ti + 3Al \rightarrow Al_3Ti \tag{13}$$

$$Ti + 2B \rightarrow TiB_2 \tag{14}$$

$$Ti + 2B + 3Al \rightarrow TiB_2 + Al_3Ti \tag{15}$$

Principles of X-ray Diffraction

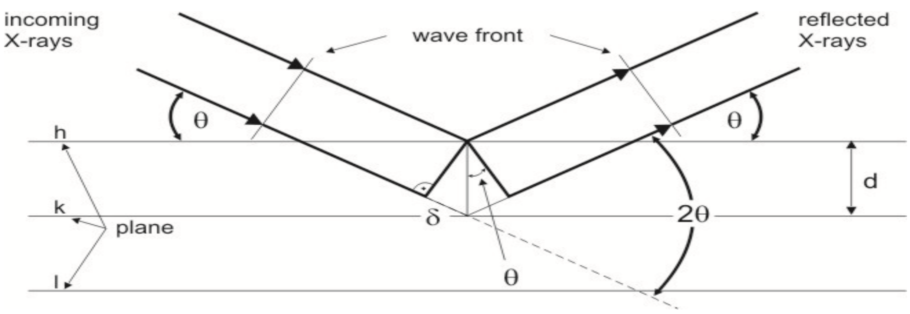


Figure 14

Where a, b, c, = Lattice constants, h,k,l = Miller Indices, dhkl = Interplaner distance/spacing, λ = wave length = 1.54, n = order of reflection=1, 2, 3, ……..… a positive integer, = Braggs angle = angle of incident.

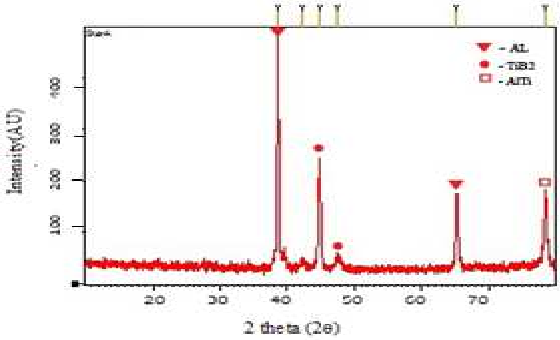


Figure 15: XRD of Al2011-5%TiB_2 Composite (As Cast before Heat Treatment)

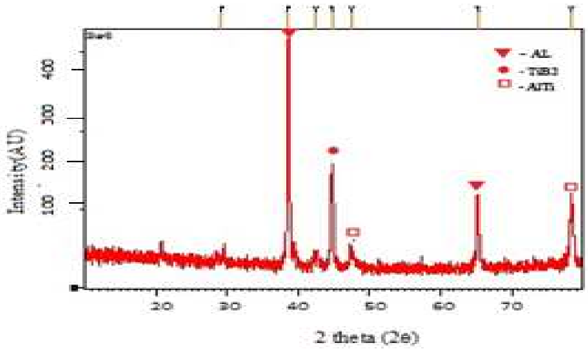


Figure 16: XRD of Al2011-5%TiB_2 Composite (As Cast after Heat Treatment)

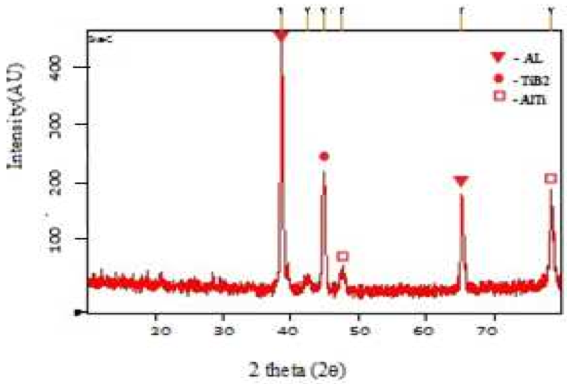


Figure 17: XRD of Al2011-10%TiB_2 Composite (As Cast before Heat Treatment)

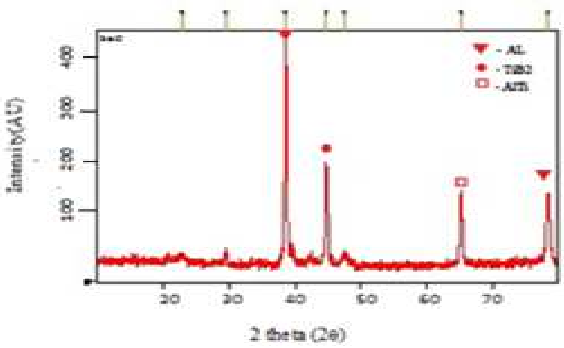


Figure 18: XRD of Al2011-10%TiB_2 Composite (As Cast after Heat Treatment)

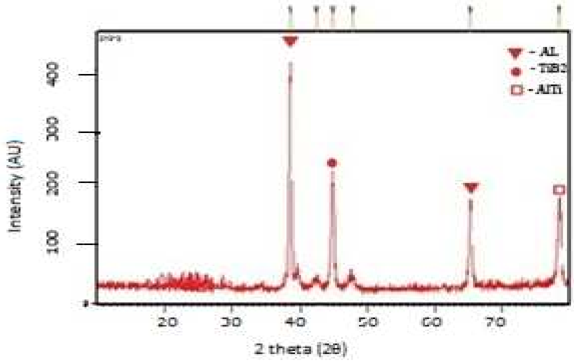


Figure 19: XRD of Al2011-5%TiB_2 Composite (Extruded before Heat Treatment)

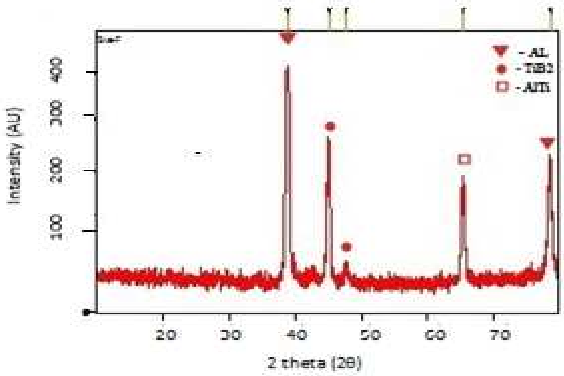


Figure 20: XRD of Al2011-5%TiB_2 Composite (Extruded after Heat Treatment)

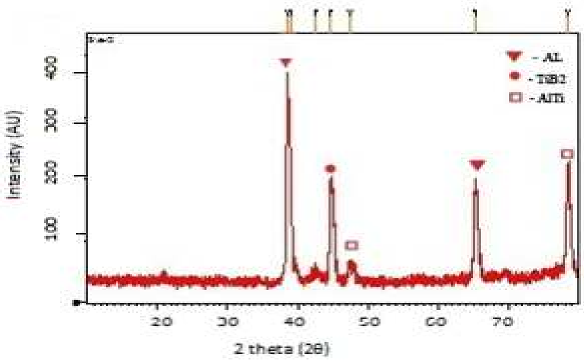


Figure 21: XRD of Al2011-10%TiB_2 Composite (Extruded before Heat Treatment)

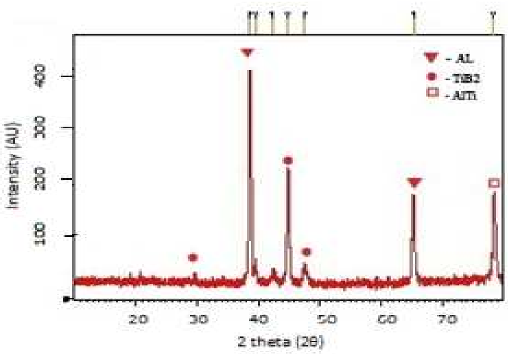


Figure 22: XRD of Al2011-10%TiB_2 Composite (Extruded after Heat Treatment)

The TiB_2 particles in the extruded composites are smaller than as cast composites. A refined grain structure is observed in extruded alloy and composite from the XRD test. Both as cast and extruded composites are free from casting defects. Further, there exist good bond between the matrix and the particle reinforcement even after extrusion composites (Foo, 1994).

3.3. Micro Hardness of Al2011- TiB_2

The average hardness of aluminum 2011 with 51 HV was specified by the addition of the TiB_2 reinforcement to the matrix, the average hardness of Al-5% TiB_2 and Al-10% TiB_2 were increased to 70 to 88 HV at 5kg test load. Hardness value of the fabricated Al-5% TiB_2 and Al-10% TiB_2 composite for 5 kg test load are given in Table 3.

Table 3: The Table for Hardness Vickers Test

Process	AL2011+ % of TiB_2	BH=Before Heat Treatment AH=After Heat Treatment	Sample Code	Value of Micro Hardness (1)	Value of Micro Hardness (2)	Value of Micro Hardness (3)	Average Micro Hardness Value (HV)
Cast	5%	BH	A	65.92	75.7	_	70.81
		AH	B	78.43	79.9	79.16	79.16
	10%	BH	C	76.19	74.20	75.05	75.14
		AH	D	81.31	79.15	_	80.23
Extruded	5%	BH	E	85.89	82.32	_	84.10
		AH	F	81.18	78.79	81.98	80.65
	10%	BH	G	92.07	86.53	86.57	88.39
		AH	H	92.32	89.17	93.01	91.5

In Vickers micro hardness test the hardness is calculated by using the equation 16. $HV=1854(F/d^2)$ (16) Where HV= Vickers hardness value

F= Indentation load in grams

d= Diagonal of the indentation in μm

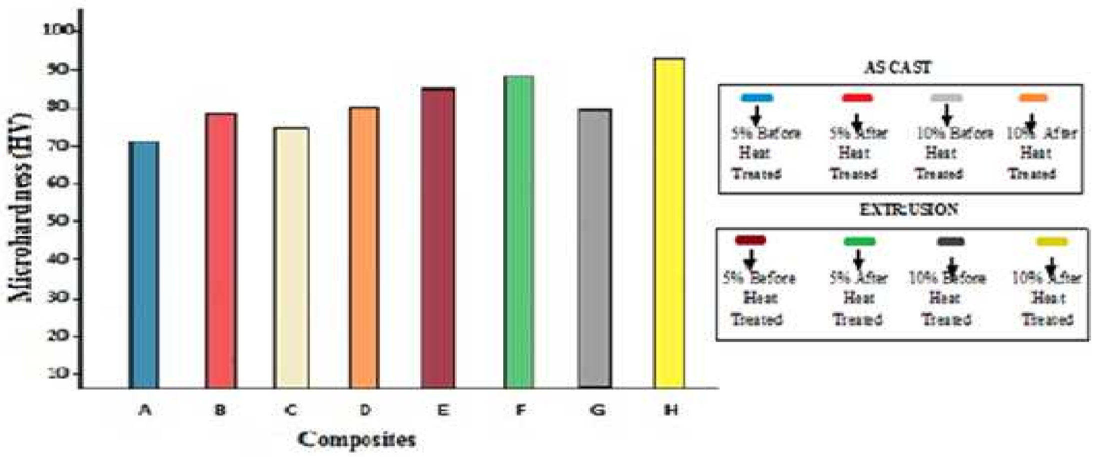


Figure 23: Comparative Values of Microhardness

In the fabricated composites the hardness value of extruded after heat treatment Al-10% TiB_2 composites is higher than the Al-5% TiB_2 composites. As the test load increases the indentation size increases and which will affect the

hardness values as shown in figure 23. Depending upon material, type of indenter and size of indentation a proper test load can be selected (Ansary Yar, 2009).

4. CONCLUSIONS

Al2011-TiB_2 composite is prepared from stir casting method using Al-5%TiB_2 and Al-10%TiB_2 master alloys. The developed composite was extruded successfully at a temperature of 500^{o}C. XRD analysis the microstructure reveals fairly uniform distribution of TiB_2 particles. After heat treated Al-10%TiB_2 extruded composite exhibited higher hardness, good corrosion limit and lower wears loss when compared with matrix alloy in both Al-5%TiB_2 as cast and extruded conditions. The incorporation of TiB_2 and Al_3Ti particles into the Al improves the corrosion resistance as compared to the unreinforced alloy. SEM and EDX microstructure analysis was carried out at 500X magnification level by using Scanning Electron Microscope. SEM analysis revealed that the particle has non uniform distribution and the EDX showed that interfacial bonding of titanium, boride and cupper is better reinforcement in the composites.

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